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POLYMERS FOR SPACECRAFT HARDWARE

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SCOPE

This report covers work performed during the period January 10, 1967 to February 9, 1967 on "Polymers for Spacecraft Hardware," SRI Project No. ASD-5046 under JPL Contract No. 950745.

The primary objectives of this program are to assist the Jet Propulsion Laboratory of the California Institute of Technology in the examination of polymeric materials to be used in connection with JPL spacecrafts, and to prove a study of the effects of simulated spacecraft environments on selected commercial polymeric products. The materials to be studied have been provided by the JPL Cognizant Engineer.

Comprehensive Polymer Test Program

The comprehensive polymer test program is designed to determine changes which have occurred in pertinent properties of polymeric materials after a decontamination treatment, a thermal-vacuum exposure, and a decontamination treatment followed by a thermal-vacuum exposure.

An entire sequence of exposures under this program was completed during this working period for 30 polymeric samples. Testing of all samples also was completed, but the data have not been entirely reduced and tabulated at this time. A detailed summary will be included in Monthly Report No. 33 for March 15.

Volatile Condensable Material

Volatile condensable material (VCM) is defined as the weight of condensate obtainable at 25° C in a given interval of time from a unit weight of a thin sample of material maintained at 125° C in a vacuum of at least 5×10^{-6} torr. The micro-VCM techniques has been established as a procedure for rapid screening of polymeric samples of the order of 100 milligrams for maximum-VCM content and total-weight-loss. The limits of acceptance for further evaluation of polymeric

products have been established as < 1% weight-loss and <0. 1% VCM, as obtained in the micro-VCM procedure.

The macro-VCM techniques, utilizing samples of 3 to 10 grams, provides information on the deposition and re-evaporation of VCM as a function of time, as well as weight-loss data; it is used for materials which qualify for further evaluation as a result of micro-VCM determinations, as well as for materials which have marginal qualifications but are unique and critical for spacecraft applications.

Macro-VCM

A series of macro-VCM determinations was just completed at the end of this reporting period. Samples of the most immediate interest are 3M's Velvet Black 101C10 (cured 160 hours at 110° C) and Mystik Tape 7452 (as received). The data obtained thus far confirm the performance expected from prior micro-VCM values, that is, a little more than 0.1% VCM for the Velvet Black and a little less than 0.1% for the Mystik Tape. The other samples comprise an experimental effort to determine the VCM behavior of known substances of differing molecular weights.

Micro-VCM

Mention has been made (Interim Report No. 3) of the need for procurement specifications and quality control to guard against batch-to-batch variations of polymeric materials which have been found to be acceptable for spacecraft use, and of the need for clear instructions for the final processing of commercial products. Another concern of spacecraft engineers is that technicians may not be apportioning 2-part polymers correctly or even may not be mixing 1-part materials thoroughly. An example of the latter is given: Because of the need for acceptable spacecraft lubricants, and since lubricated areas are not as likely to become as warm as electronic component areas, it was agreed with the JPL Cognizant Engineer that lubricants may be screened at the lower temperature of 70° C. Thus, two Electrofilm lubricants were re-screened at 70° C, and also re-determined at 125° C for

comparison. It was found that Electrofilm 4306 qualifies for spacecraft candidacy (<1% wt-loss, <0.1% VCM) and Electrofilm 2396 approaches candidacy at 125° C (see Table I). Thus, these new values supercede those reported in Interim Report No. 3 as 3.19 and 1.37% wt-loss and 0.87 and 1.25% VCM, respectively. Examination of the lubricants revealed that solids were heavily deposited in the containers and required mixing beyond that even of a mechnanical paint mixer; apparently the first VCM data were obtained primarily for the solvent and binder with little solids content. As shown in Table II, there is little significant difference between weight-loss and VCM at 70° C or at 125° C.

Micro-VCM determinations are generally made on materials which have been cured according to manufacturer's instructions or after more stringent cures based on experience (e. g., silicones, polyurethanes). However, other cures are often used in practice, and at the suggestion of the JPL Cognizant Engineer, a number of materials are being rescreened after the treatments dictated by prevailing practice at JPL. As shown in Table III, VCM values for a number of protective coatings were unacceptable after the stringent cure of 24 hours at 150° C; after the milder cures suggested by JPL, the materials are still unacceptable as far as VCM is concerned, and have incurred greater weight losses.

Table IV is a summary of data on two new sealants; Hysol C7/4248 does not qualify, but Hysol 5150/3690 may, after a more stringent cure.

The possibility was discussed with JPL Cognizant Engineers some time ago of extending the micro-VCM determinations so as to provide a rapid and diagnostic method for quality control by using infrared spectrophotometry to assess the presence, nature, and quantity of VCM. Weight loss still is determined in the procedure, but the weighed copper collecting discs for VCM are replaced by optical salt flats 1" diameter and 1/16" thick. The VCM is collected on the salt flats which are maintained at 25° C for 24 hours as before. Then, the salt flats are examined in the infrared region from 2-15 microns.

In the preliminary runs, the salt flats were weighed to ensure that they were adequately cooled to collect the maximum VCM per sample weight as was collected in duplicate determinations using the weighed copper plates. As shown in Table V, the pick-up of VCM on the salt flats is not significantly different from that collected on copper plates. The general tendency toward a slightly lower %-VCM is attributed to loss of moisture from the salt flats and an occasional loss of salt from spalling caused by clamps.

Representative infrared spectra of the VCM from some of these materials are given in Figures 1 to 3. Further development of this method will include the establishment of absorbance limits per sample weight (not calculated at this time), and the preparation of reference spectra for the most acceptable materials. Additionally, retainers will be designed to eliminate spalling and provide accurate measurement of weight of VCM.

In Monthly Report No. 21 it was reported that the wt-loss and VCM of RTV-41/T-12 (GE) could be improved to acceptable values by a post-cure of 24 hours at 250° C, but it appeared that physical properties were degraded. In order to check these observations, freshly-prepared materials were postcured at room temperature for 24 hours at 150° C, and for 24 hours at 250° C, and subjected to actual mechanical properties tests. As shown in Table VI, the ultimate tensile strength drops sharply after the 150° C cure, and remains about the same after a 250° C cure; elongation does not vary very much after the 150° C cure, but increases significantly after the 250° C cure; Shore hardness drops drastically after the 150° C cure, and slightly more after the 250° C cure.

FUTURE WORK

Work will continue on micro- and macro-VCM determinations for polymeric materials. Identification volatile materials will be made.

Equipment for 8-month storage tests for mechanical properties will be refurbished for future runs.

Work will be initiated for Run No. 3 in the Comprehensive Polymer Test Program.

Table I

Micro-VCM Determinations: Lubricants

(24 hr at 125° C and 10^{-6} torr)
(VCM collector plates at 25° C)

Material	Mfr.[1]	Treatment	Total Wt.[2] Loss, %	VCM,[2] wt-%
Silicate-MoS_2-G				
Electrofilm 2396	EFI	Cured 2 hr/80° C plus 2 hr/205° C	0.32	0.20
Electrofilm 2396	EFI	Postcured 16 hr/205° C	0.21	0.16
Phenolic-MoS_2				
Electrofilm 4306	EFI	Cured 1-1/2 hr/190° C	0.72	0.20
Electrofilm 4306	EFI	Postcured 16 hr/190° C	0.67	0.09

[1] EFI, Electrofilm, Inc.

[2] Values supercede those reported in Interim Report No. 3.

Table II

Micro-VCM Determinations: Lubricants

(24 hr at 70° C and 10^{-6} torr)
(VCM collector plates at 25° C)

Material	Mfr.[1]	Treatment	Total Wt. Loss, %	VCM, wt-%
Electrofilm 2396	EFI	Cured 2 hr at 80° Cplus 2 hr/205° C	0.21	0.09
Electrofilm 2396	EFI	Postcured 16 hr/190° C	0.20	0.09
Electrofilm 4306	EFI	Cured 1-1/2 hr/190° C	0.63	0.12
Electrofilm 4306	EFI	Postcured 16 hr/190° C	0.56	0.15

[1] EFI, Electrofilm Inc.

Table III

Micro-VCM Determinations: Protective Coatings;
Effect of Cure Cycles
(24 hr at 125° C and 10^{-6} torr)
(VCM collector plates at 25° C)

Material	Mfr.[1]	Treatment	Total Wt. Loss,%	VCM, wt-%
Epoxy				
Stycast 1217/9	EMC	Postcured 24 hr/150° C (SRI)	0.57	0.12
Stycast 1217/9	EMC	Cured 16 hr at 52° C	1.74	0.14
Polyester-phenolic				
Eccocoat PH-7	EMC	Postcured 24 hr/150° C (SRI)	1.55	0.84
Eccocoat PH-7	EMC	Cured 2 hr at 50° C	5.22	1.92
Polyimide				
Pyre-M. L. RK-692	DUF	Cured 24 hr/150° C (SRI)	3.00	0.48
Pyre-M. L. RK-692	DUF	Cured 1 hr at 95° C plus 1 hr/150° C plus 1 hr/205° C	1.34	0.35
Silicone				
SR-220	GES	Cured 24 hr/150° C (SRI)	3.52	2.86
SR-220	GES	Cured 5 hr at 150° C	3.76	2.71

[1] EMC, Emerson and Cuming, Inc.
DUF, E. I. duPont de Nemours and Company, Fabrics and Finishes Department
GES, General Electric Company, Silicone Products Department

Note: "SRI" Cure cycles were adopted to reduce VCM and weight loss; the values were included in Interim Report No. 3 and are reproduced here. The other cure cycles reflect current practice at J. P. L.

Table IV

Micro-VCM Determinations: Sealants

(24 hr at 125° C and 10^{-6} torr)
(VCM collector plates at 25° C)

Material	Mfr. [1]	Treatment	Total Wt. Loss, %	VCM, wt-%
Epoxy				
Hysol 5150/3690	HYS	Cured 24 hr/25° C	1.82	0.17
Hysol C7/4248	HYS	Cured 2 hr/25° C plus 16 hr/175° C	0.66	0.23
Hysol C7/4248	HYS	Postcured 24 hr/150° C	0.74	0.18

[1] HYS, Hysol Corporation

Table V

Comparison of VCM Pick-Up on
Optical Salt Flats vs Copper Collectors

(24 hr at 125° C and 10^{-6} torr)
(VCM collectors at 25° C)

Sample	Wt-% VCM, Salt Flat	Wt-% VCM, Copper Disc
Eccocoat PH-7	2.08	1.92
SR-220	2.71	2.44
RTV-60 (24/150° C)	0.54	0.34
Eccocoat IC-2	0.29	0.32
Stycast 1217/9	0.14	0.12
Hysol 5150/3690	0.17	0.08
Viton A4411A-778	0.08	0.01
Teflon FEP 100A	0.08	0.04

Table VI

Effect of Curing Cycles on Mechanical Properties of RTV-41/T-12 (General Electric)

Cure / Sample	Test Length		Ultimate Tensile Strength		Elongation, %	Shore Hardness, Type A
	Before	After	lb	lb/in²		
24 hr at 25° C						
A	1"	2.2"	6.8	340	120	
B	1"	2.5"	6.5	325	150	
C	1"	2.5"	6.3	315	150	
D	1"	2.75"	8.1	405	175	
E	1"	3.00"	9.2	460	200	
Average				369	160	57
24 hr at 150° C						
A	1"	2.4"	5.0	236	140	
B	1"	2.4"	4.6	217	140	
C	1"	2.6"	6.0	283	160	
D	1"	2.75"	6.9	325	175	
E	1"	2.3"	5.0	236	130	
Average				259	149	48
24 hr at 250° C						
A	1"	3.0"	5.7	285	200	
B	1"	2.7	3.9	195	170	
C	1"	3.25"	6.2	310	225	
D	1"	3.1"	6.1	305	210	
E	1"	3.0"	5.2	260	200	
Average				290	201	47

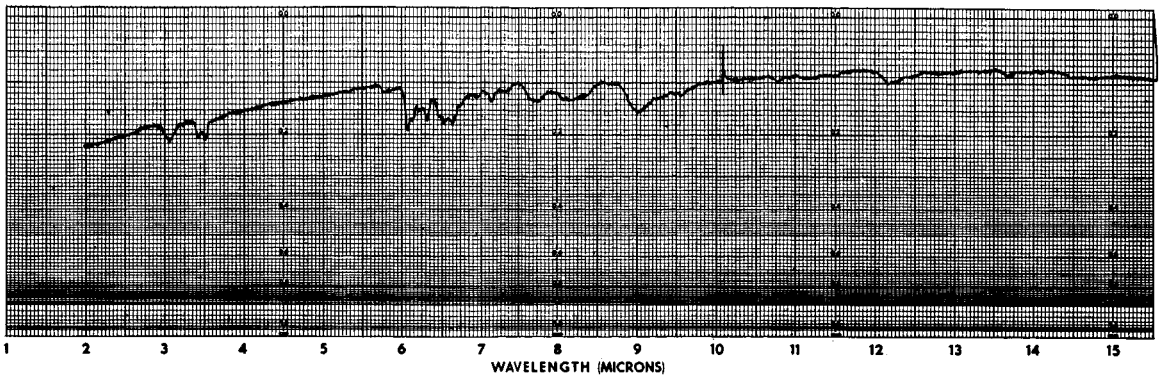

FIG. 1 INFRARED ABSORBANCE SPECTRUM OF VCM FROM ECCOCOAT IC-2
(about 80 mg sample, about 0.3% wt-% VCM, cured 8 hr/121 °C)

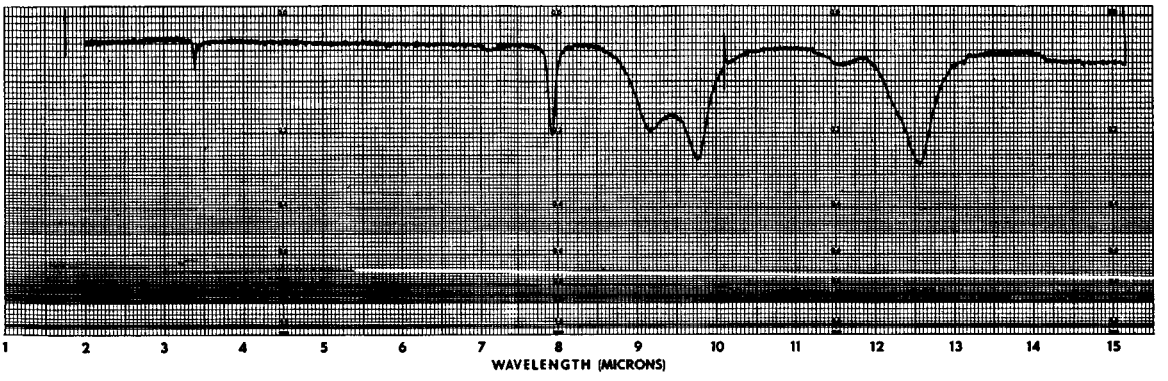

FIG. 2 INFRARED ABSORBANCE SPECTRUM OF VCM FROM RTV-60/T-12
(about 50 mg sample, about 0.5 wt-% VCM, sample postcured 24 hr/150 °C)

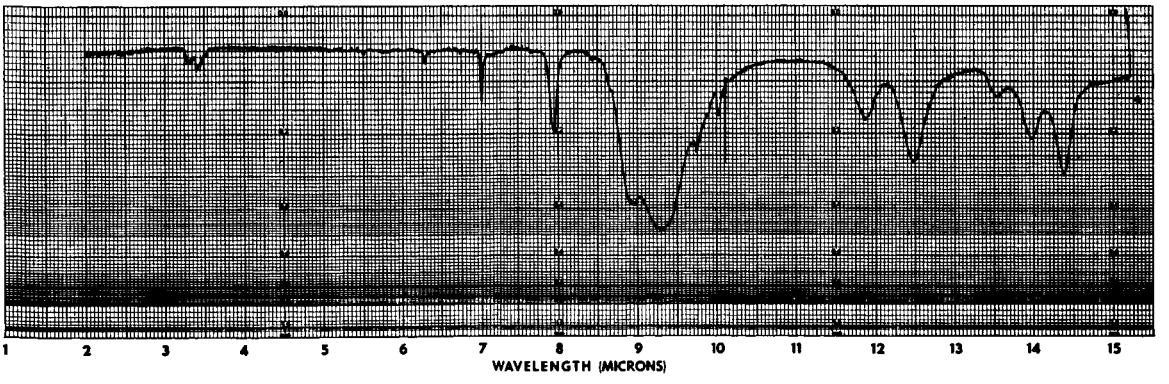

FIG. 3 INFRARED ABSORBANCE SPECTRUM OF VCM FROM SR-220
(about 15 mg sample, about 2.7 wt-% VCM, sample cured 5 hr/150 °C)